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(NASA-CR-171163) SURFACE ANALYSIS OF SPACE TELESCOPE MATERIAL SEECIMENS Monthly Report (Auburn Univ.) 70 p HC A04/MF A01 CSCL 03A N84-34351

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SURFACE ANALYSIS OF SPACE TELESCOPE MATERIAL SPECIMENS (NAS8-35914)

Monthly Report for July (July 31, 1984)

Albert Thomas Fromhold, Jr. Department of Physics Auburn University, AL 36849



Prepared for

George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812

Abstract

Surface analysis by electron spectroscopy for chemical analysis (ESCA) was used to characterize a number of the samples during the past month. With ESCA, the sample is irradiated with monoenergetic soft x-rays and the resulting emitted electrons are energy analyzed to determine the binding energy of electrons to the surface atoms. The major peaks were used in the quantitative determination of the surface composition. (Please refer to Tables 1 through 16 which are interspersed with the figures.) The presence of trace elements (impurities below 1% atomic composition) was also detailed. Initially a survey scan was run for each sample to deduce the elemental composition. Then the major peaks of interest and those of the trace elements were individually examined. After this, the samples were argon sputtered to etch away surface layers, and then additional measurements were carried out in order to obtain depth profile information. Those species present only on the surface could in this way be distinguished from those having a significant depth distribution within the sample.

It was estimated on July 31 that the measurements were 90% complete. It is expected that the measurements will be 100% complete by August 30, 1984.

ESCA Data for Silver and Copper Specimens

Silver

Four silver samples have thus far been examined: a silver interconnect flown on STS-8, a silver foil flown on STS-8, the relevant control silver specimen for these samples, and a gold-plated silver foil which had been subjected to the plasma asher for a period of two minutes. Several common trends were found for all silver samples:

- 1. The flight and ashed samples exhibited a slight decrease in binding energy (0.1 to 0.3 eV) of the Ag 3d doublet relative to the control. (These data are given in Figs. 1, 2, 9, 21, and 22.) These results indicate an increase in the degree of oxidation of silver (viz., Ag ==> $Ag_2O ==> Ag_3O$).
- 2. Chemical shifts toward oxygen species with lower O(1s) binding energies (B.E.) occurred in the flight and ashed samples. (These data are given in Figs. 5, 6, 11, 12, 17, 18, 23, and 24).
- 3. The control sample always had a Ag to O ratio of 1:1 at the surface of the sample. (These data are given in Tables 2, 4, 6, and 8. It should be pointed out, however, that this is not necessarily evidence that the chemical composition of the surface is AgO, although the data are consistent with this interpretation.)

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4. A decrease in carbon was observed in the ashed and flight samples relative to the control. (See Tables 1, 2, 3, 4, 5, 6, 7, and 8.)

The ashing process, surprisingly, did not seem to simulate very well the processes which occurred during exposure to the ambient during flight. The flight samples exhibited shifts toward increased surface concentrations of oxygen at lower binding energies, but new chemical bonding forms were not observed. Figures 17, 18, 23, and 24 indicate only shifts between oxygen species originally present on the surface. On the other hand, the ashed samples exhibited not only this effect, but also the creation of new oxygen species at still lower binding energies (see Figs. 5, 6, 11, and 12).

Ashed and flight samples also differed in the silver to oxygen ratio. Whereas the control showed a 1:1 ratio, the ashed samples showed a decrease and the flight samples showed an increase in this ratio. The results are as follows:

Silver-to-Oxygen Ratios

Control Sample:	1.0
Gold-plated Silver after Ashing:	0.2
Silver Foil after Ashing:	0.4
Silver Foil after Flight:	2.5
Silver Interconnect after Flight:	1.5

The gold-plated silver sample deserves special mention due to the unusual effect of the plasma asher treatment. Even the control sample exhibited a silver peak at the surface, which is rather surprising. (It would be expected that the gold plating would have entirely protected the underlying silver.) The ratio of gold to silver was found to he 4 to 1. After ashing, gold was barely detectable (see Figs. 3 and 4). Quantitatively it was found that the gold to silver ratio went from 4 to 1 before ashing to a ratio of 1 to 13 after ashing. Possibly this could have been due to a bulk diffusion of the gold into the silver foil during ashing. It seems unlikely that the gold was sputtered away, since the sample weight measurements by the Marshall personnel indicated no weight loss during ashing. Another possible explanation is that the adhesion of the gold to the silver was affected by plasma ashing, so that the gold aggregated into small beads, thereby leaving the silver surface exposed.

The composition of the silver interconnect and the underlying silver foil after flight were very similar (see Tables 6 and 8). However the control sample for the interconnect was found to have a larger amount of graphitic carbon. It was found that this carbon could be easily removed by sputtering. The flight interconnect had somewhat more carbon than the flight foil. The flight foil also contained sulfur and chlorine, elements not not found on the interconnect.

Copper

Cupric oxide was found on both the control and flight samples (see Fig. 27). The amounts of carbon and oxygen were less in the flight samples, as indicated below.

Oxygen-to-Copper and Carbon-to-Copper Ratios

	0/Cu	C/Cu
Control Sample:	5.2	5.9
Flight Sample:	1.1	3.8

A shift to lower binding energy oxygen was observed in the flight sample (see Fig. 28). The carbon type, however, was unchanged (see Fig. 29).

ESCA Data for Paint Specimens

Because the paint samples were non-metallic, some electrical charging occurred during the ESCA study. consequence, the positions of the peaks at times appeared as much as 6 eV higher than expected. This was more evident in the A-276 and the 401-C10 specimens than in the Z-302 specimen, the latter being scarcely affected by charging. The degree of surface charging depends not only on the low electrical conductivity of the sample, but also on the thickness of the specimen because this will control the rate of charge leakage to the underlying metal substrate. The consequence is that peak positions were inconsistent from sample to sample, and in addition, the peak positions were observed to change with sputtering of the sample. Therefore the energy axis of the ESCA scans should not be used to infer absolute binding energy, and the shifts in energy should not be used indiscriminantly to infer changes in chemical binding.

In view of the foregoing, it is not surprising that few consistent trends were found by means of ESCA for the paint specimens. Nevertheless, the A-276 and Z-302 samples did show a slight decrease in the silicon-to-oxygen ratio, while the 401-C10 sample exhibited a significant increase. The data supporting these statements are now given.

Silicon-to-Oxygen Ratios

	A-276	Z-302	401-C10
Control Sample:	0.48	0.29	0.13
Flight Sample:	0.30	0.19	0.88

For the A-276 specimens, the silica, oxygen, and carbon peaks were unaffected by flight, as indicated by comparison of the flight and control sample data (see Figs. 31, 32, and 33). For the Z-302 specimens, the silica peak was broader in the flight sample, and the peak was broadened by sputtering (see Figs. 34 and 35). No change occurred in the N(1s) or O(1s) peaks (see Figs. 36, 37, 40, and 41). Three forms of carbon were present on the Z-302 control. The flight sample exhibited a shift in one of the higher binding energy forms of carbon (see Figs. 38 and 39).

Although the peaks in the 401-C10 flight sample appeared to be shifted towards higher binding energy, this seems to be a consequence of a difference in the degree of charging of the flight and control specimens. Ferhaps the thicknesses of the samples were not the same. Assuming this to be the case, there does not appear to be a difference in the oxygen species between the flight and control specimens (see Figs. 42 and 43). Two forms of silicon were present on the control specimen, with the higher binding energy form predominant. The flight sample, on the other hand, showed no lower binding energy form (see Figs. 44 and 45). The carbon peaks indicated a behavior resembling that of the Z-302 samples, namely, the flight sample exhibits a shift to a higher binding form relative to the control sample (see Figs. 46 and 47.

Sample: Gold-plated Silver / Ashed z min Treatment: Date: 7-31-84

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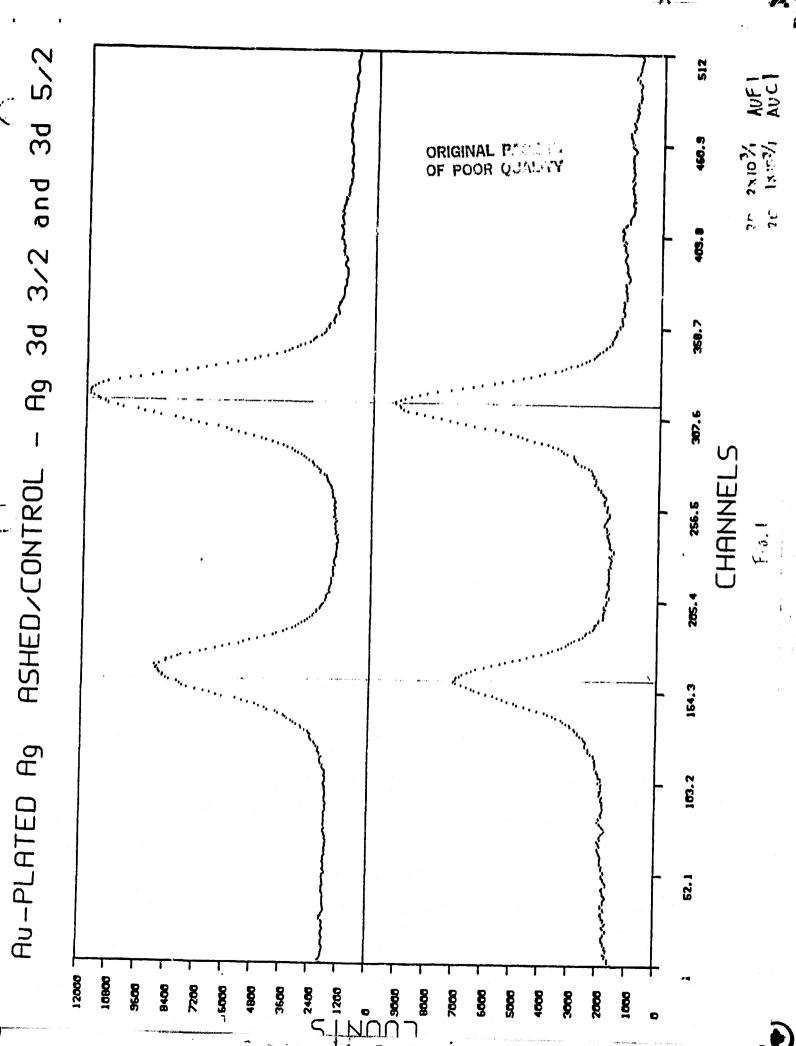
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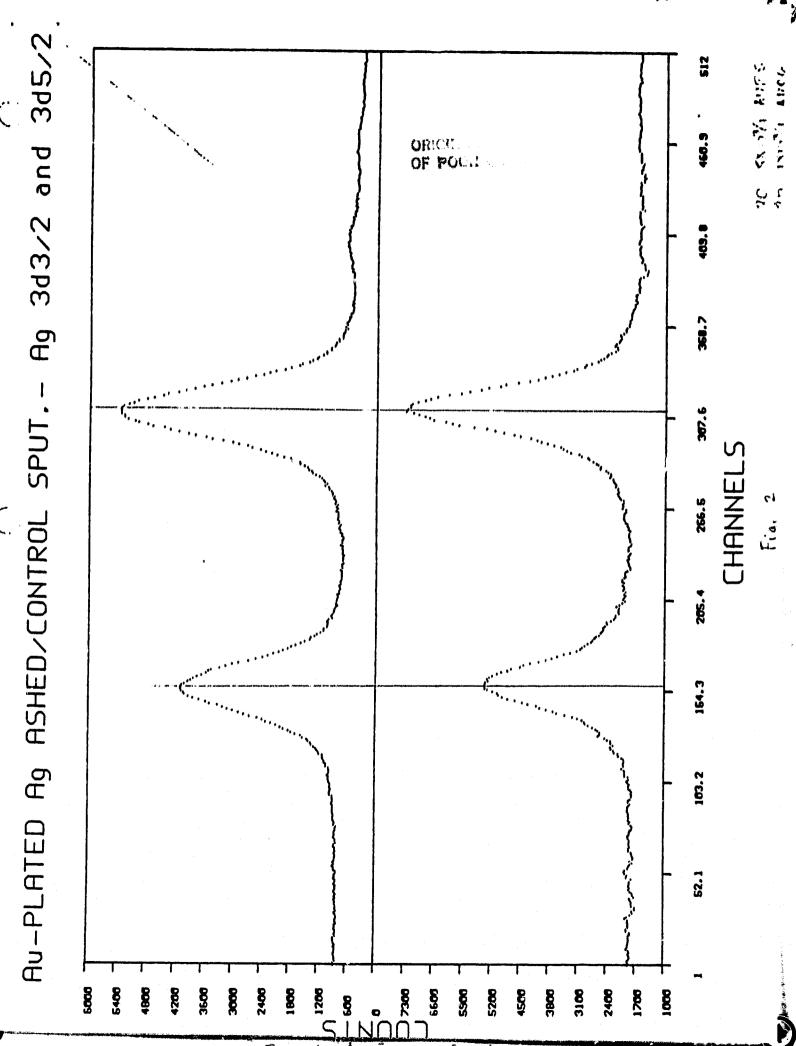
mole frac,

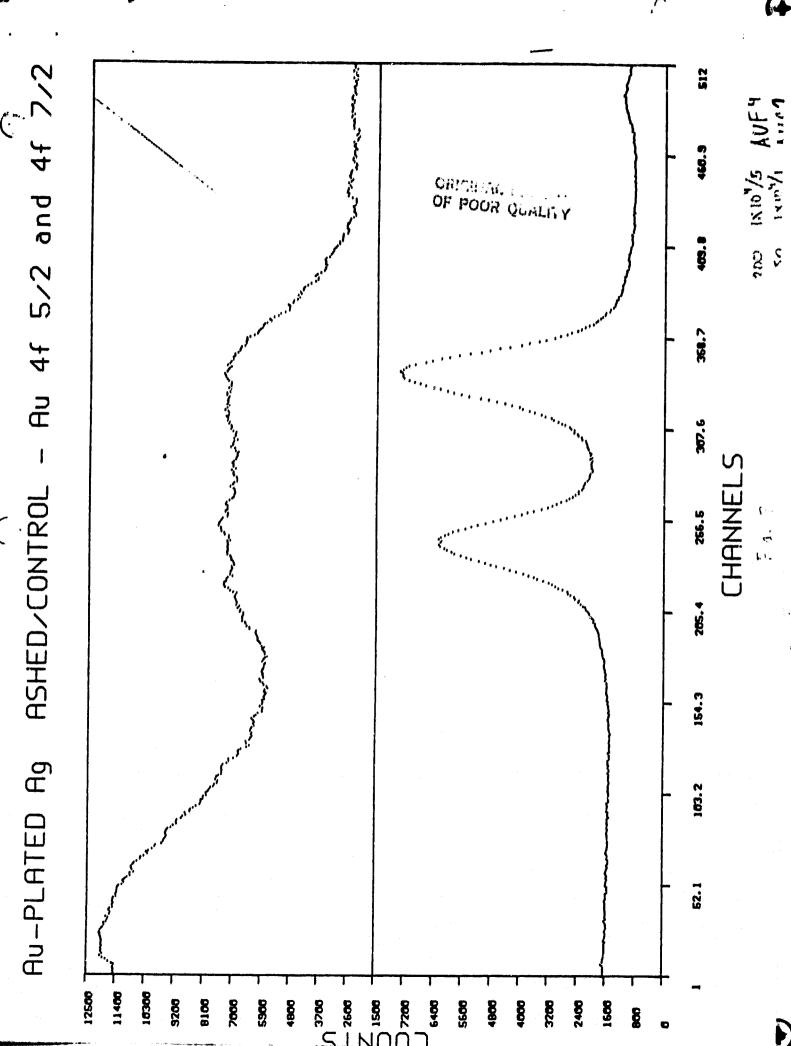
Sample: Gold-plated Silver /Control Treatment:
Date: 7-29-84

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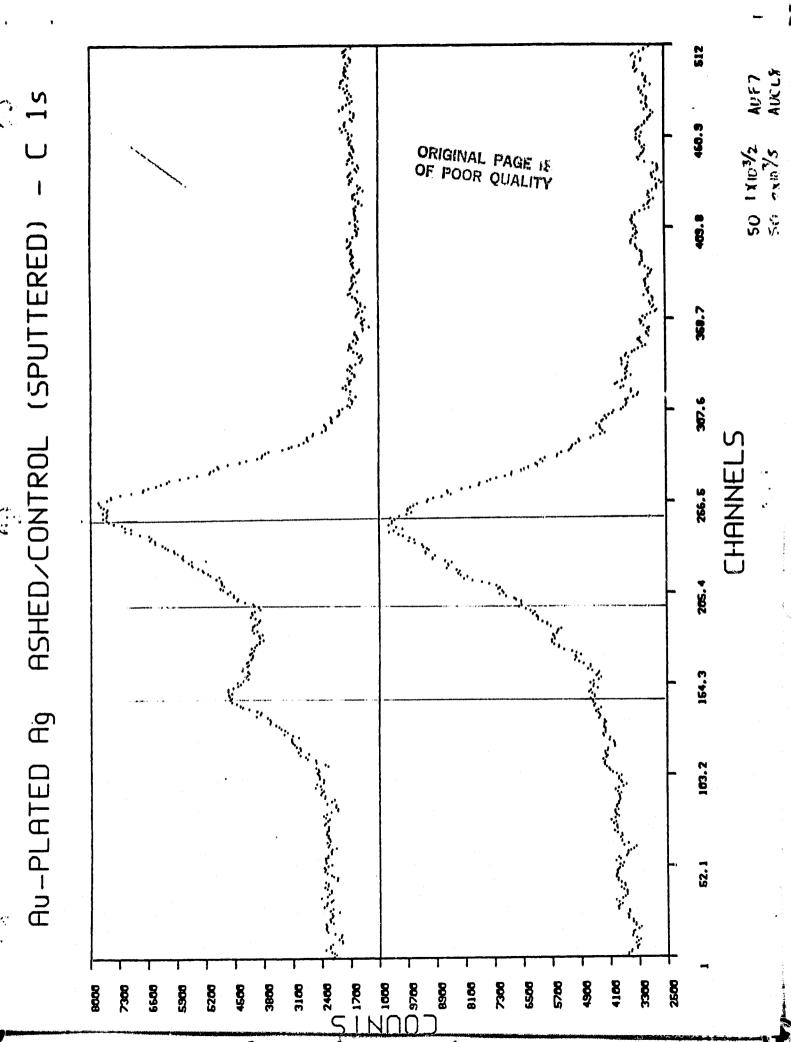
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٠. ج		310.7 7X103		282	333			175.7		
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peak	deAbor Agal s.	desder	•	AGA601 0(15)				(31)		-
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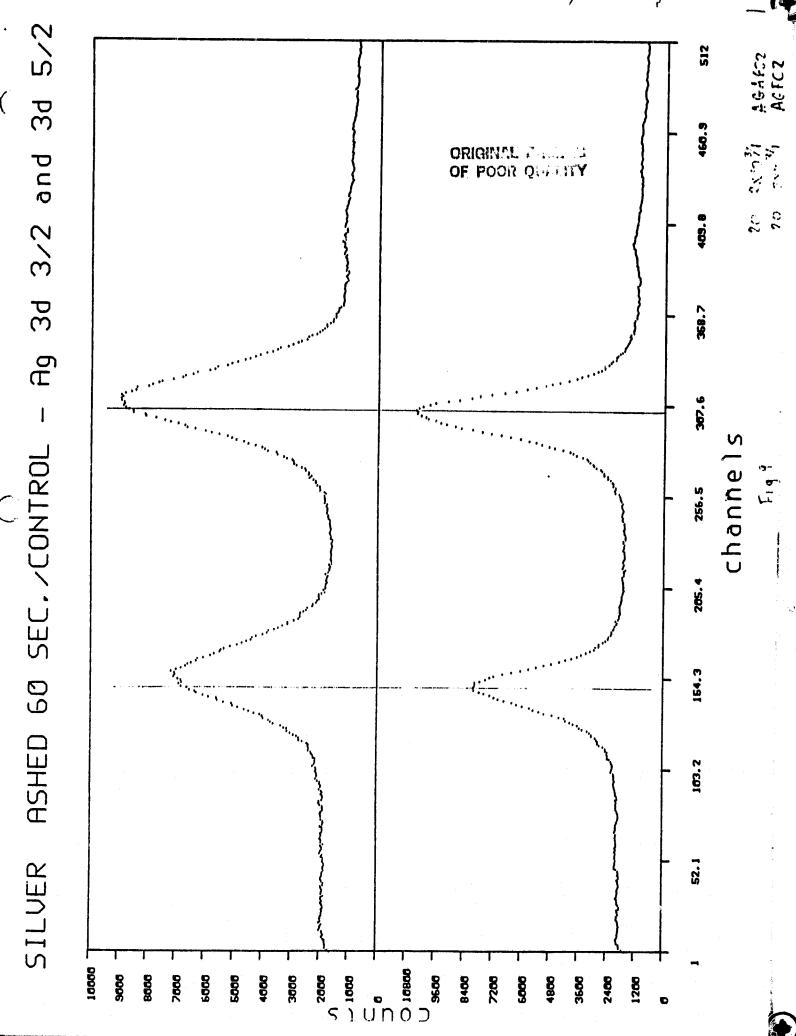
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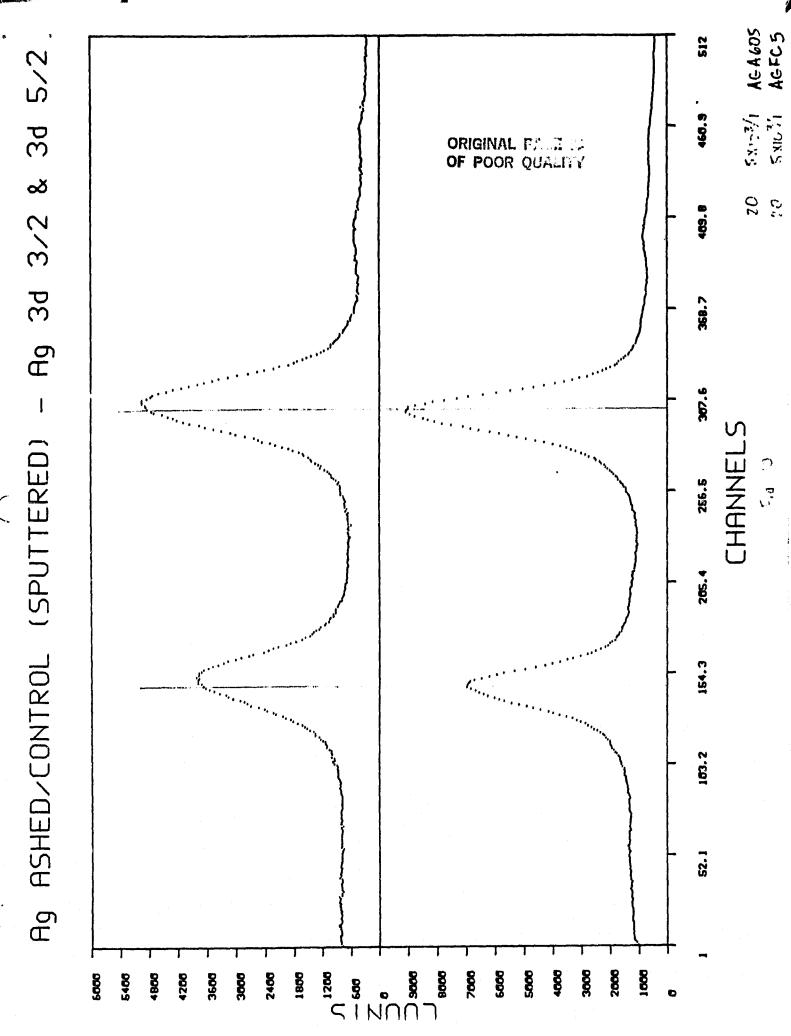
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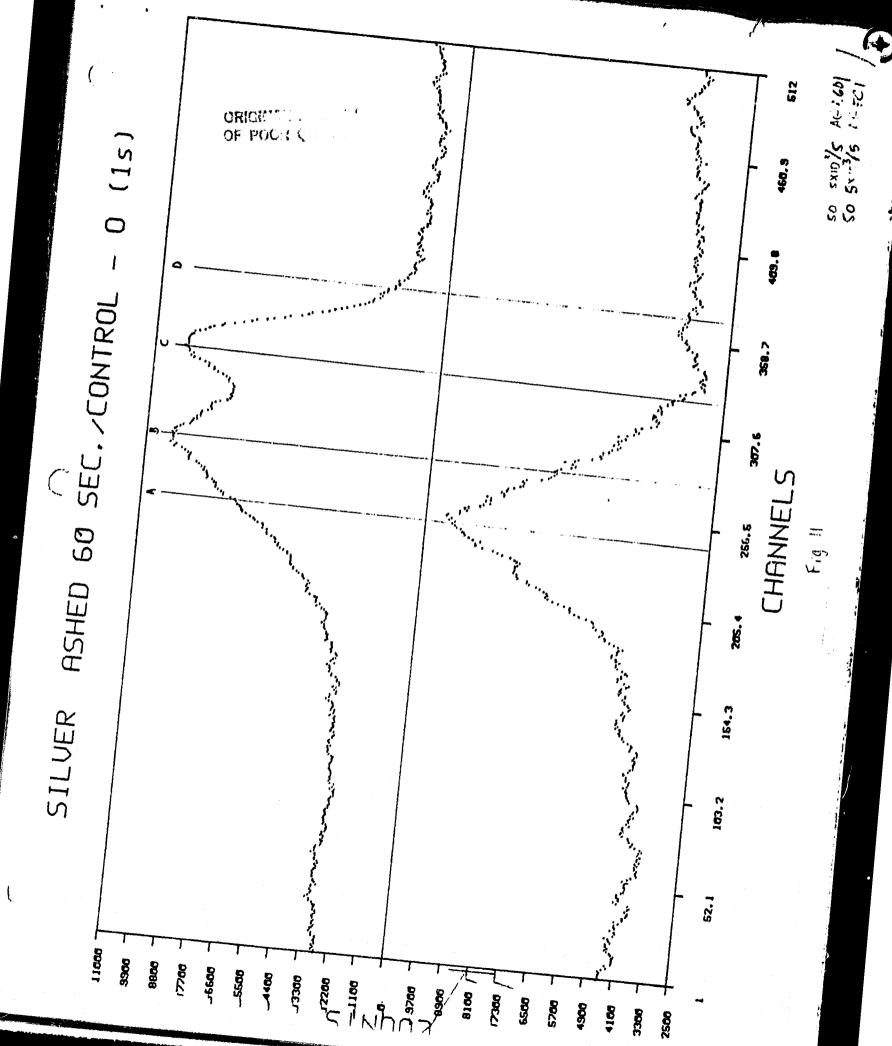
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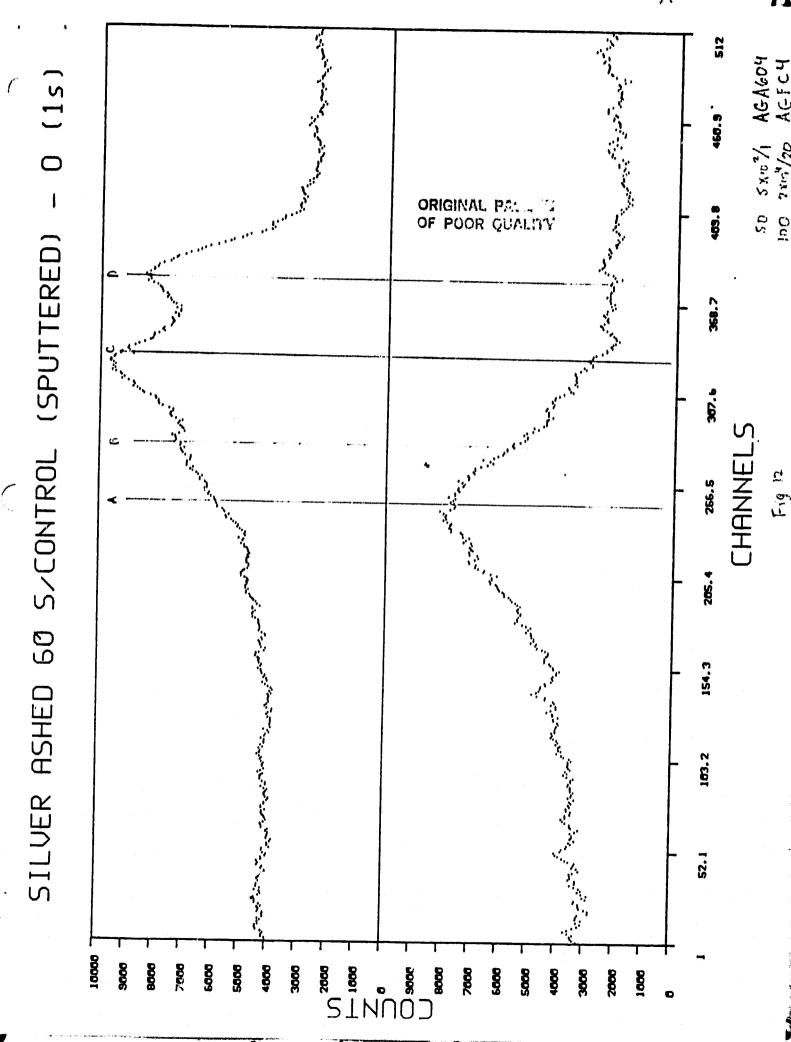
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Sample: STS-8 Treatment: Date: S-1-¢4

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mole frac.		0.40			,	0.16			hh.o	
cross sect.		10,7		2.85			1.00			
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E _O (eV)	50			50			20			
inten.	2 x 10 1/1			s/¿alxs			5/501x8 S115-1			
ch. #		308		152	Lb7			1	-	
8.E. (ev)	374	398		532.2	530,4		C (10) 286.9	284.3		
peak	A 36 36	As 30 54	``	(36)6						
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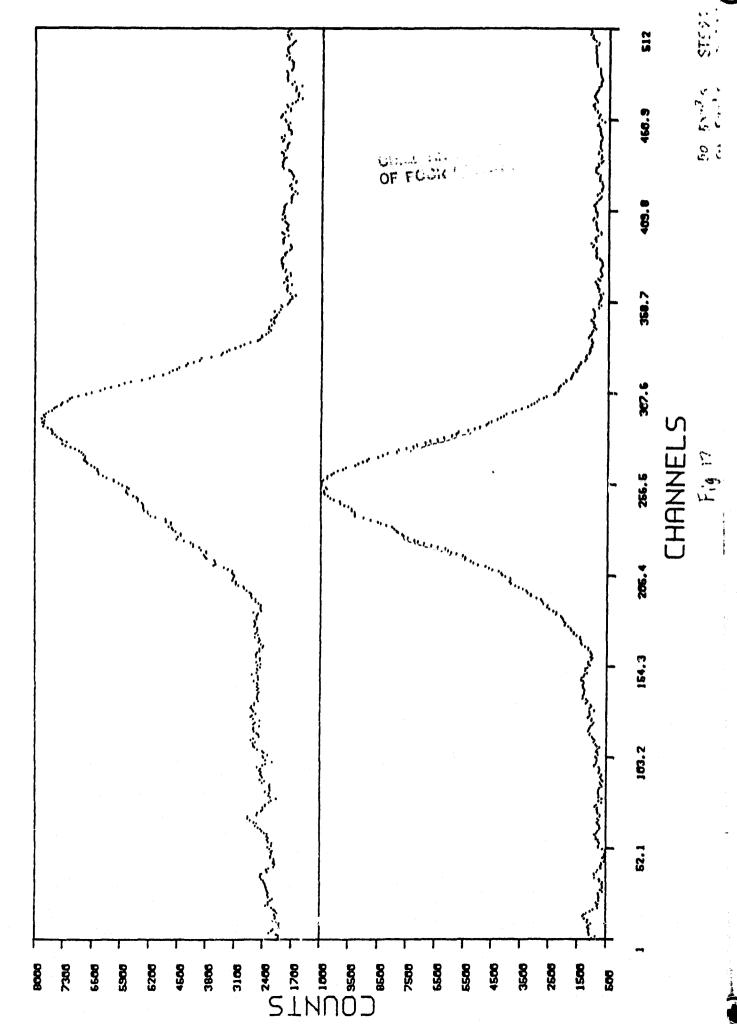
Sample: STS-8 Confiel Treatment: Date: R-1-84

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mole frac.		0.19		2.85 6.20	,				170	io :0	
cross sect.		10,7 0.19		2.85			1.00	200			
escape depth		15 %		148			1 x	1771			
norm. area		273.000 S. 46 x 10 15 Å		726,000 1.454x10 14 R					332,000 1.46 x107		-
area		273.000		726,000					332,000		_
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E ₀ (eV)	50			22			So				
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ਦ		308		532.2 251			188.7	238.7			
8.E. (eV)	hLE	378		532.2		1	7%2.1	284.7			
peak	7861 A. 20m	10 MG		(N)0			2156 C(18/ 28.1 188.7 SXID%				
Scan	1.3523			176(1			2/3/			-	

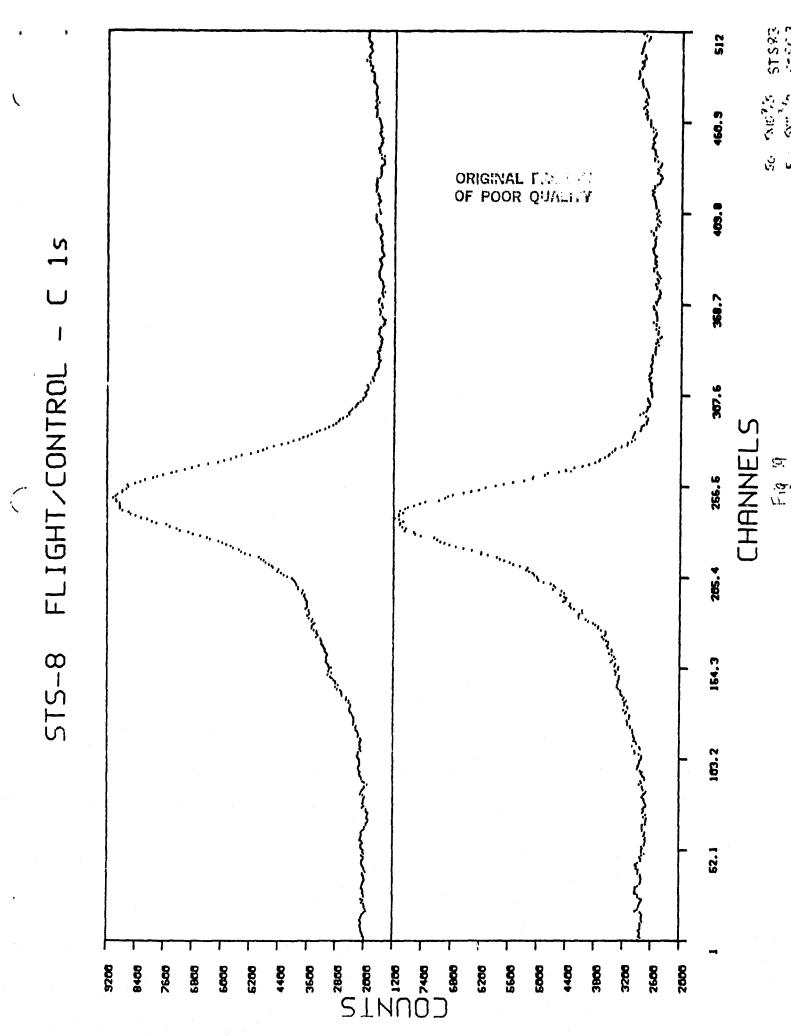
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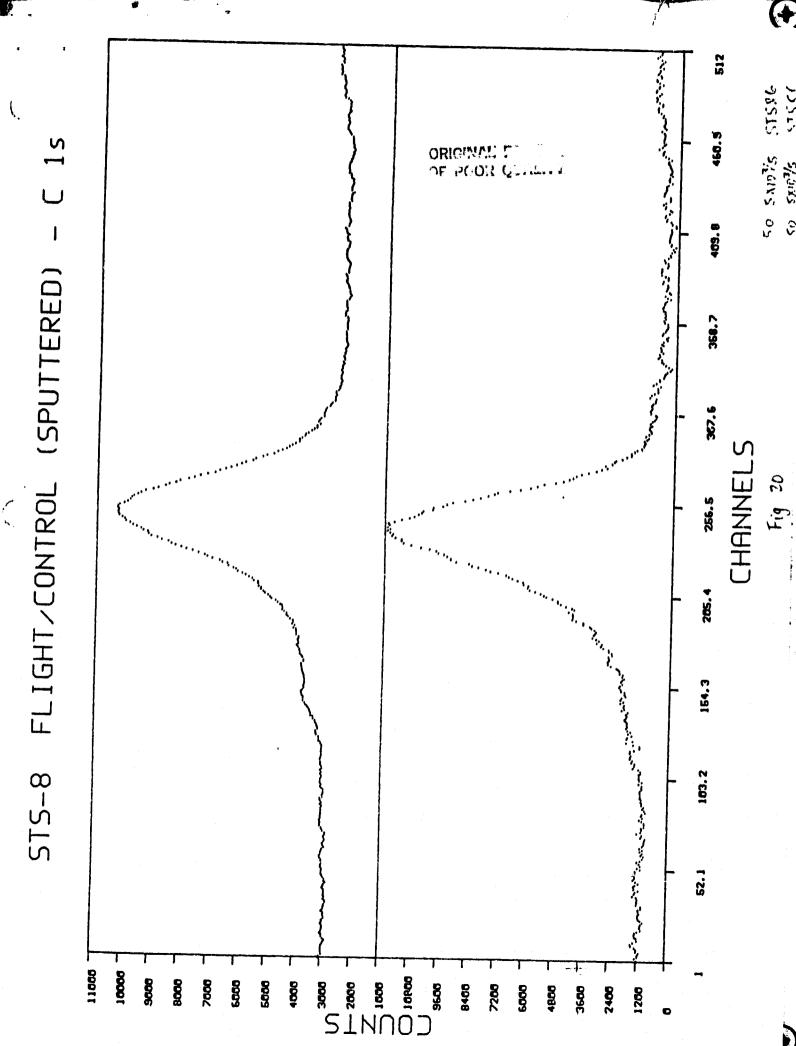
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FLIGHT/CONTROL (SPUTTERED)





Sample: Silver of Flight Treatment: Date: 7 84

		Ag					0				7	
mole frac.		0.22				,	0.15				0.63	
cross sect.		10,7		285				1.00				
escape depth		233,000 4.66x107 15 A		14 A				15 A				
norm. area		4.668107				30.00	1014 634,000 1.: 110				613,000 1,226×10	
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inten.	NOW.			532.2 251.4 5x102/2 20			16	275.7 213.1 5x10 /5				
ch.		312.7		751.4	297.4			213,	247.6			
Β.Ε. (¢۷)		A. 24 55 367. 2			530.4				1,045 247.6			
peak	An 32 32	N. 24 55		AG S 0(18)			17./-	(3/)				•
scan	AC 3			マウン			11 0 2	1 -117				

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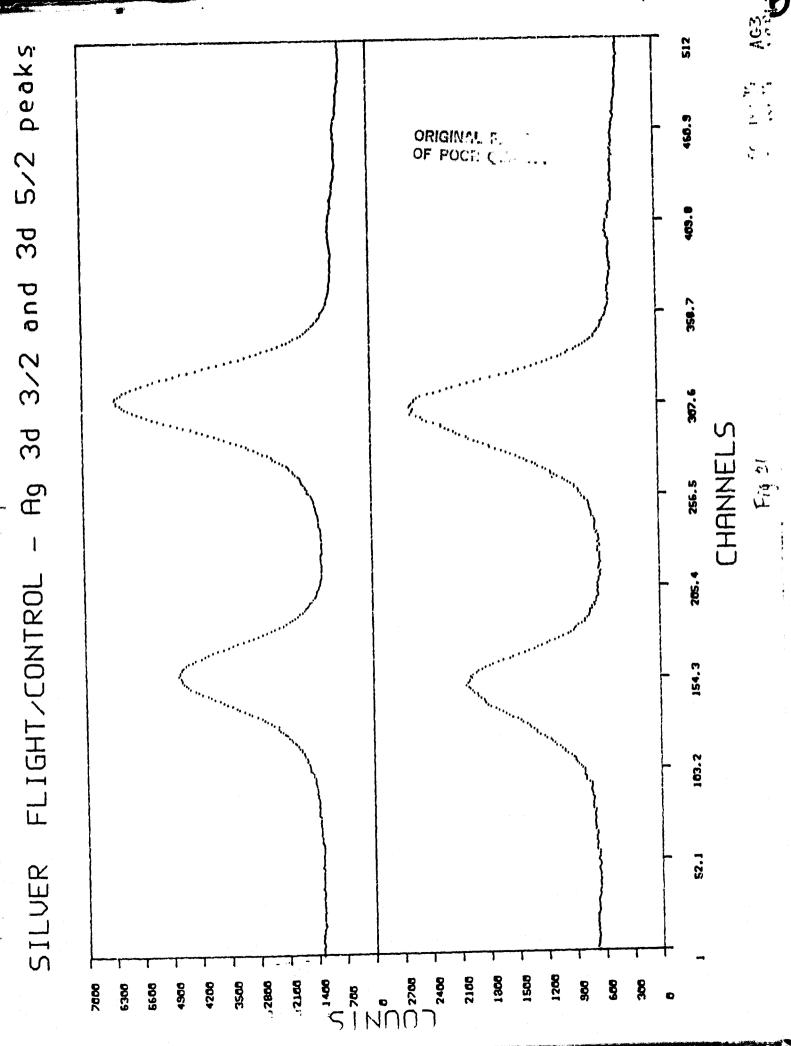
Table 8

Sample: Silver /Confrol Treatment: Date: 7-27-84

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mole	frac.		71 0							2 0			010
cross	sect.		10.7			2.85				091			
escape	depth		KA	1. 21		M				IS A			-
norm.	area		97.500 1. 15x10 15 B					ח סטנ אינו	1.563AIV				7 98 XID
area			47.500					JULY DOO IL ONE YIN	286,000				John 1299 north 98x10
E ₀ height	(counts)		2005			2008	2000	10.401	10.1				10/01
F ₀	(ev)	05				2.0			(80			
inten.		Ka vi			1	251.4 Sx10/2			۲	्ण्			
ch. #			305		:	251.4	797.4			220, 1 5 1/10	246.3	287.2	
8.E.	(ev)		As 5156 36x.1 305			532.2	C 30. 4			285.4	284.4	282.8 287.2	
peak		AGREE No Edition	A4 54 5/2		10,70	AGBLS UCIST 539.2			1	AC AC! ((18) 285.4			
scan		AGREE				ACBCS			A. P. C. L.	AC N			

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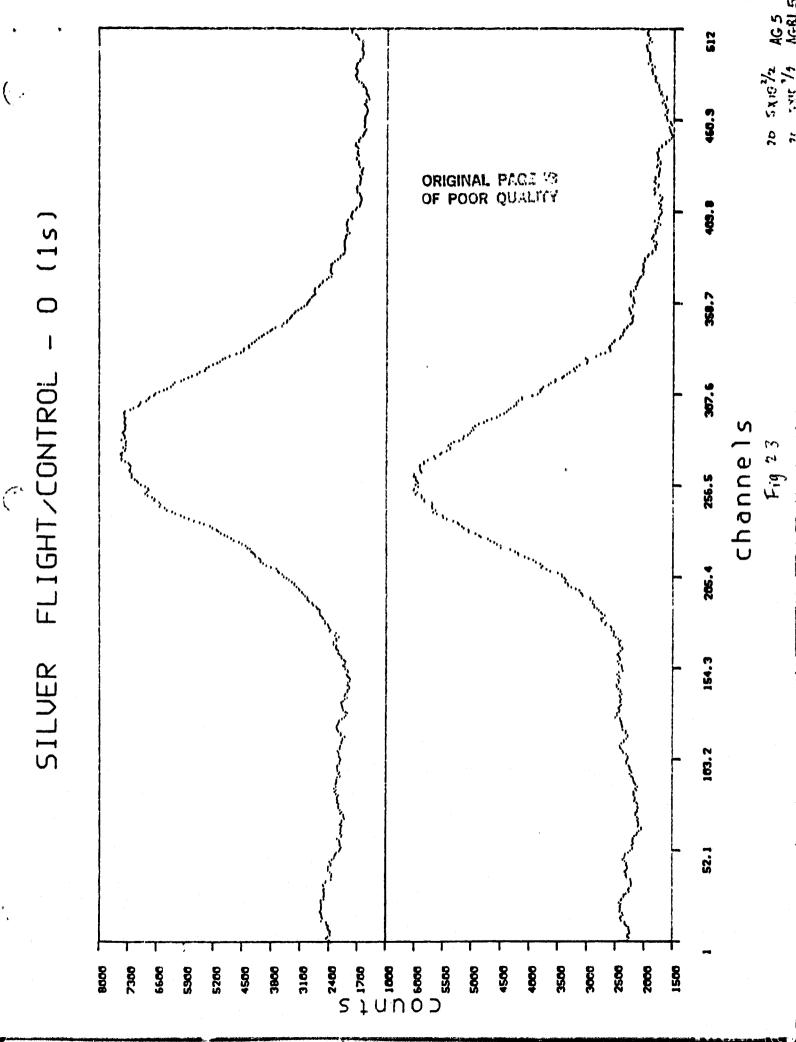


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					Çu		Q		2
mole frac.					.6.17		6 19		h9.0
cross sect.	∞ 		15.9		24.08 .C.17	554		(,0)	
escape cross depth sect.	10			-		h l		51	
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area	1,072,00	•				1,009,00			 16,900
E _o height (eV) (counts)				- .•		,		NSA (x3)	
E ₀ (eV)	001					50		991	
inten.	5/h0125					₹011 S		5×1071	
ch. *									
8.E. (¢V)									
peak	Cu 203		() 2p 3;			(si)O		C(IS)	
scan	CUY					200		200	

Sample: Copper /Flight Treatment: Date: 7-19-84 ORIGINAL PARENTY
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018750.E 030,214 117,025 total 001 100

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.0.683 24.08 cross sect. 2.85 18.9 100 5,13 escape depth Ξ 0 <u>د</u> 835,00c 8.35 x107 7.17 x 107 norm. area area height (counts) total (eV) 100 5/h "x 2 13.10 1/1 1/2 01 X 3 irten. ch. 🖈 Sample: Caper Conford Treatment: Date: 153.6 452.2 B.E. (eV) 933,6 10 213 Cv 2p 2 (3:) peak (R) (UPL Y CUBLE :' scan ر.

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0.43

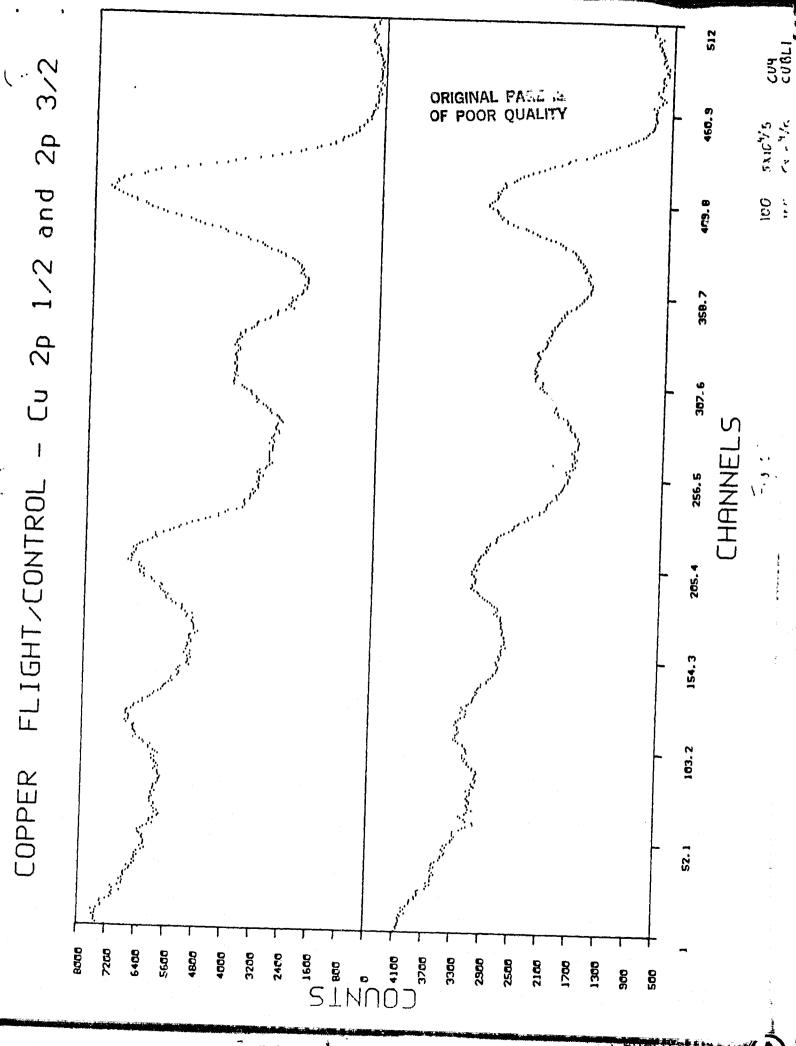
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Sample: A - 276 Flight Treatment: Date:

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mole frac.	01.0	01.0		0:41									_
cross sect.	010 7/30	2 80	1.0										
escape depth	- 1	14 B	ISA							•			
norm. area	1152 11.520	1176 11760	1500 15 000 15 A	2227			,						_
area	7511.	1176	1500									-	
height (counts)				-	•		,						
E ₀ (eV)	So	So											
ch. # Inten:	52102/1	Sx103/1	1 1/163/2										
			-										
8.E. (eV)	109.3	538.7	291										
peak	A276A S.(25) 109.3	0(18)	A1766 ((15) 291					,					
scan	AZTEA	AzzeB	A276C										

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mole frac. 0.33 0.16 0.51 0.865 2.85 cross sect. -:0 escape depth <u>-</u> S 85,250 028,02 14,030 norm. area 1705 1258 1403 area height (counts) (eV) SC 80 20 5×103/2 5×1071 2x103/1 inten: ch. # 105.2 287.5 535.3 8.E. (eV) Si (2p) (Si) O (R) peak A2764 A 2763 A 2761 scan

Sample: A-276 Control Treatment: Date: 8-8-84

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A276 FLIGHT/CONTROL - C 1s

Table 13

Sample: 2—300 | Flydil Treatment: Date:

	ζ.	0	ر ر					
mole frac.	0.05		29'0		٠			
cross sect.	6,865	5.85	1.0					
escape depth	15 A	4-1-	13					
norm. area	P182 F185	1236 49,440	1195 47,80c			•		
area	J882.	1236	1195					
E ₀ height (counts)				-		٠		
E ₀ (eV)	200	So	ΰŚ					
inten:	SYON	2 × 15 /1	1/201.KG					
ch.								
B.E. (eV)								
peak			1					
scan	72	7 222	2 30 6					

Comments:

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Tall . 14

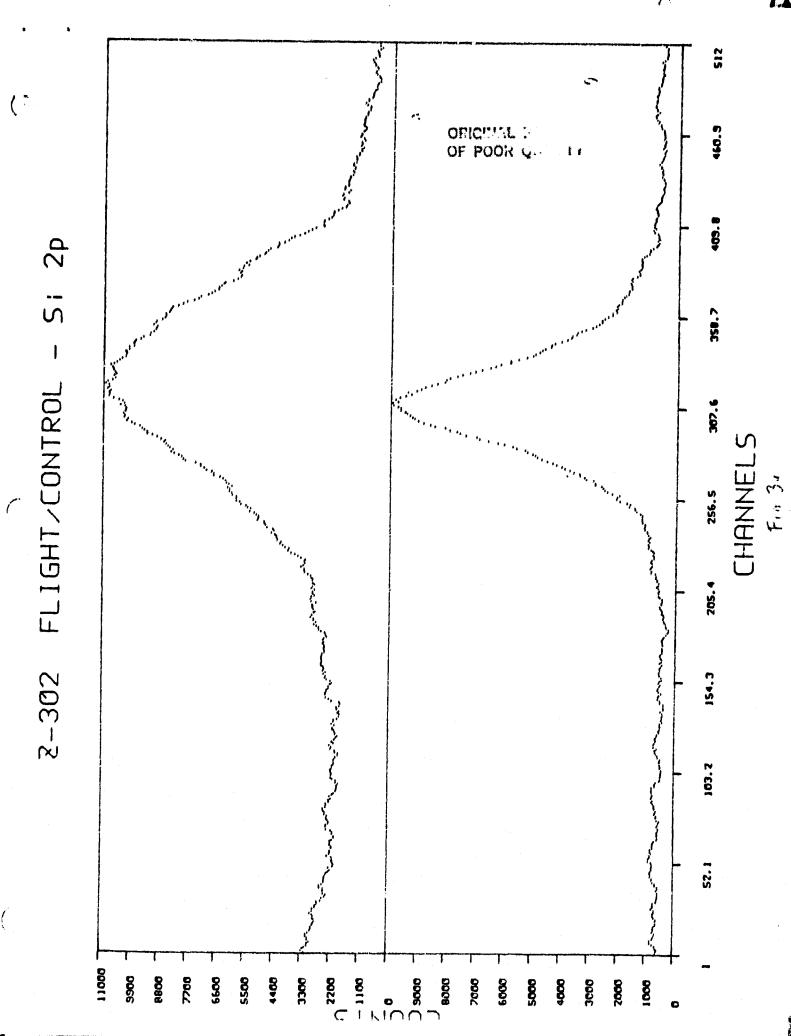
Sample: 2-302 (coth.) Treatment: Date:

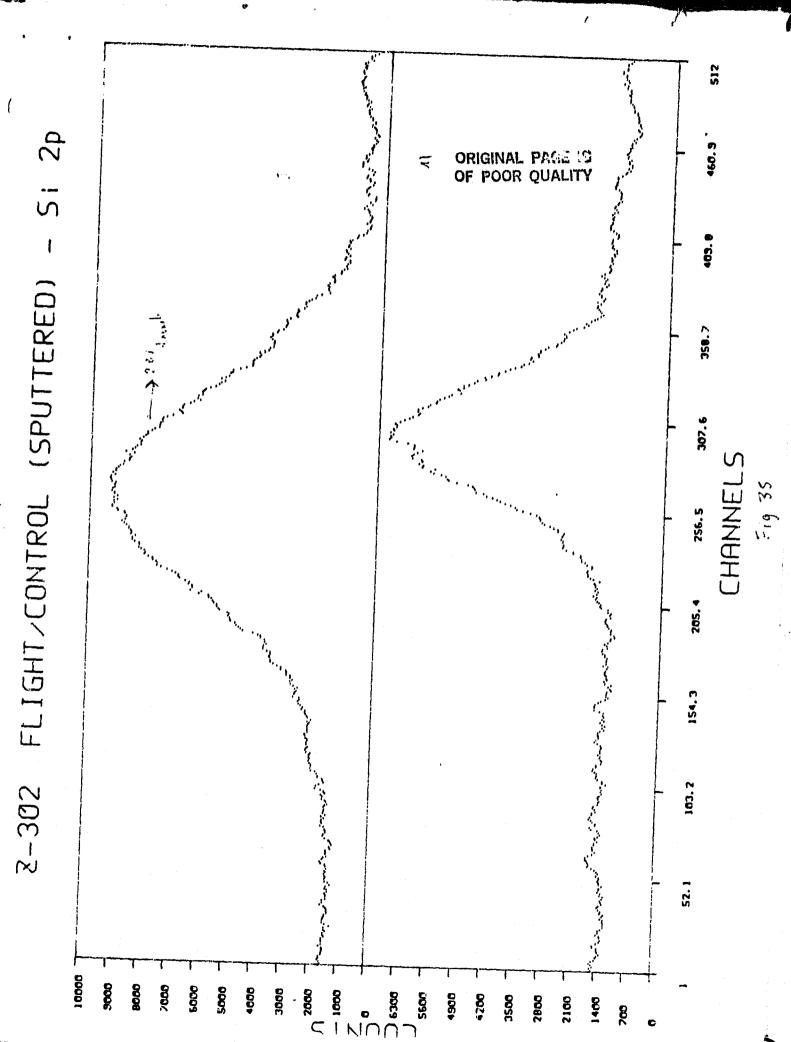
T	T	1	Т-			 				<u></u>	
	٠.٪	C	U					•			
mole frac.	0.05	71.0 35.	1,01 0.72								
cross sect.	6.865 0.05	36 4	1,71								
escape depth	15	5 1	5								
norm. area	1685 5370	875.5 35,co	1548 61,900								
area	5891.	1875.5	1548								
height (counts)				•		,					•
(eV)	- 1	500	56								
inten.	27 283	1/201×2	2 × 10-7/1								
ch. //				-			-				
B.E. (eV)	133.9										
peak	1015	- 1	C(11)								
scan	2302A	23C2.B	27:10								

Comments:

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OF POOR QUALLA





2-302 FLIGHT/CONTROL - 0 1s

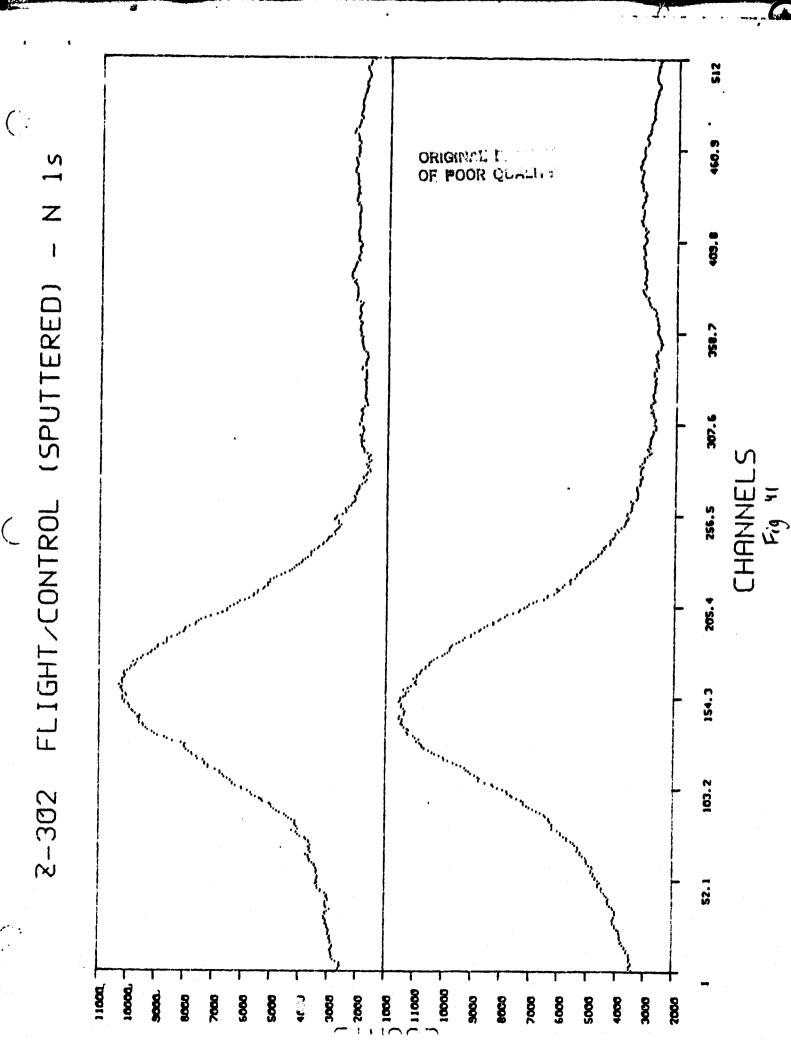


Table 15

0.10 0.42 mole frac. 84.0 0,865 5.8.2. 0 cross sect. escape depth 15 AP <u>h</u>.. 157,500 1236 44,720 12,380 norm. area 7542 1575 area height (counts) е (e У 20 50 50 5x103/1 5x102/2 1/ ¿arx I inten: ch. B.E. (eV) 1. . . . (-1) (31) peak .C.11. 7 לוט ג scan 210

Sample: "Joj-60 Flyhd Treatment: Date:

Comments: low Sileyel

ORIGINAL : OF POOR Q

Since Mailly Cast

Table 16

mole frac. 0.04 0,32 79.0 . 2.95 0.865 cross <u>·</u>0 sect. escape . depth 15 A Ы.. 79.820 60,400 3464 norm. area 1988 1510 1732 area height (counts) е СО СО 20 55 200 2 N103/1 2x103/1 inten: 1/2 01/1 ch. B.E. (eV) (31)0 5:(21) (iv) peak CIDB CIOC Y JIJ Scan

Sample: $uOI - CIO^{-l}Ol^{-l}I$ Treatment:

Date:

Comments:

ORIGINAL FA

5;, 5,6,1

512 (460.9 ORIGINAL PARTICIPATION OF POOR QUALITY 409.8 401-C10 FLIGHT/CONTROL (SPUTTERED) 358.7 307.6 CHANNELS Fig 43 256.5 20S.4 Thistory 154.3 103.2 52.1 1.09800 , 00801 12000 6000 36.00 2400 - 1200 3600 2200 2400 8400 7200 4800 3600 8400 6000 4800 3600 1200

Si 2p 410-C10 FLIGHT/CONTROL

410-C10 FLIGHT/CONTROL